

PATENT SPECIFICATION

NO DRAWINGS

965,901



965,901

Date of filing Complete Specification (under Section 3 (3) of the Patents

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PATENTS ACT, 1949

In

SPECIFICATION NO. 965,901

In accordance with the Decision of the Superintending Examiner, acting for the Comptroller-General, dated the 19 June 1968 this Specification has been amended under section 14 in the following manner:-

Page 6, delete lines "6 and 7"

Page 6, for Claims "7 to 16" read "6 to 15" inclusive

Page 6, line 12, for "7" read "6"

Page 6, line 16, for "1 to 8" read "1 to 7"

Page 6, line 20, for "1 to 9" read "1 to 8"

Page 6, line 30, for "1 to 11" read "1 to 10"

Page 6, line 35, for "12" read "11"

Page 6, line 41, for "10" read "9"

Page 6, line 55, for "15" read "14"

Page 6, delete lines "59 to 64"

Page 6, line 68, for "18" read "16"

Page 6, line 86, for "20 or 21" read "17 or 19"

Page 6, for Claims "18 to 22" read "16 to 20"

25 PATENT OFFICE,
4th September 1968

D 106708/7

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elevated temperature, the liquid medium comprising for example oils or water soluble liquids such as glycerol. It has further been proposed to effect a preliminary gelatinisation in a water-soluble liquid and then to effect the final gelatinisation in the same liquid by raising the temperature of the liquid to more than 160° C.

the reaction in the plasticiser is too slow. At temperatures much above 200° C. decomposition occurs. Suitable substances which can be used as a liquid medium in the method of the present invention include vinyl polymer plasticisers such as tritolylphosphate, polyhydric alcohols such as ethylene glycol, propylene glycol and glycerol, aromatic hydroxy compounds such as resorcinol, tertiary

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Index at acceptance: —B5 A(1R14C1X, 1R20, 1R27, 1R29X, 1R100, 7A); A3 V(1A1C1, 7B1); B2 B(4E1A1, 4E1A3, 4E1C1, 4E2A, 4E27, 4E3D, 4E5A, 4E6A, 4E7AY, 4E7BX, 4E8D, 4E8E, 4E9P, 4E9Q2, 4E9QY)

International Classification: —B 29 d, f (A 41 d, B 0 5)

COMPLETE SPECIFICATION

Improvements in or relating to the Manufacture of Protective Clothing

We, ROBERT. EDWARD CHAPMAN, a British Subject, of 23, Westella Way, Kirkella, Hull, Yorkshire, and GEOFFREY PARKER WADDINGTON, a British Subject, of "Southlands", Old Road, Welton, Brough, Yorkshire, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the manufacture of articles from polyvinyl chloride.

Articles such as gloves have previously been produced by dipping a preformed lining into a plastisol of polyvinyl chloride and then heating the coated lining to a fusion temperature in an oven. Attempts to manufacture articles such as unlined gloves and gloves having non-woven linings supported by the resin itself have not proved highly successful, however, in that the time required in that process for dissolution of the resin particles of the plastisol in the dispersant therefor is not of sufficiently short duration to prevent running of the plastisol on the former and parting of any non-woven lining applied thereto.

It has previously been proposed to pre-gel (i.e. partially gel) the coating or coatings applied to a former by immersing the coated former in a liquid medium at an elevated temperature, the liquid medium comprising for example oils or water soluble liquids such as glycerol. It has further been proposed to effect a preliminary gelatinisation in a water-soluble liquid and then to effect the final gelatinisation in the same liquid by raising the temperature of the liquid to more than 160° C.

It is an object of the present invention to provide a process in which the dissolution time is markedly reduced.

According to the invention a process is provided for the production of an article, especially a glove, from vinyl polymers and copolymers containing at least a major proportion of combined vinyl chloride which comprises applying to a former in the shape of said article one or more coatings of a dispersion of one or more of said polymers in a plasticiser therefor and thereafter effecting complete gellation of said coating on said former by directly contacting said coating with a liquid medium maintained at a temperature at which gellation of said dispersion takes place.

The vinyl polymers preferably consist substantially wholly of vinyl chloride. Copolymers of vinyl chloride with e.g. vinyl-acetate or vinylidene chloride may also be used, the vinyl chloride content of such copolymers being preferably at least 95%. A mixture of polyvinyl chloride with small amounts of other vinyl polymers may also be used.

The gelling temperature should be above 150° C. and may, for example be in the range of 150° C. to 200° C. preferably 160° C. to 180° C. Below 150° C. dissolution of the resin in the plasticiser is too slow. At temperatures much above 200° C. decomposition occurs. Suitable substances which can be used as a liquid medium in the method of the present invention include vinyl polymer plasticisers such as tritolylphosphate, polyhydric alcohols such as ethylene glycol, propylene glycol and glycerol, aromatic hydroxy compounds such as resorcinol, tertiary

amines such as triethanolamine and long chain unsaturated acids such as oleic acid. Compounds which have a marked solvent, swelling or chemical action on the ingredients of the plastisol should of course be avoided.

It has further been found that when the liquid medium is composed of glycerol, ethylene glycol or triethanolamine, the product obtained has a markedly increased elasticity which in some cases approaches that of rubber.

The dispersion of vinyl polymers is preferably a plastisol. Organosols or modified plastisols may be used which include volatile organic solvents as diluents but in that case it is advisable to allow the volatile solvents to evaporate from the coating before gelling is effected in the liquid medium. The plastisols are dispersions of vinyl polymers in a plasticiser therefor. The resin particles are relatively insoluble in the plasticiser at room temperature but at elevated temperatures, e.g. above 150° C, they are soluble in the plasticiser.

When a plastisol is heated to a temperature of 150° C or more, therefore, the resin particles of the dispersion dissolve in the plasticiser to form a single continuous phase.

The plastisols are mixed in shearing-type rather than high-speed mixers and at room temperature (preferably below 90° F). Stabilisers and colour pigments are usually pre-dispersed in plasticiser before being added to the plastisol. Because of the relatively viscosity of plastisols air entrapped during mixing fails to escape and must be removed for most applications by subjecting the plastisol to a vacuum.

The plasticiser may be, for example, dibutyl phthalate, di-isooctyl phthalate, or tri-tolyl phosphate. Suitable stabilisers are, for example, a calcium zinc soap, calcium stearate and epoxidised oils.

The rate of dissolution of the polymer particles in the plasticiser depends on the temperature. In the case of vinyl polymers containing a major proportion of vinyl chloride it is necessary to use a temperature of at least 150° C in order to achieve a sufficient speed of solution. The gelled material which is obtained above 150° C appears to be identical with plasticised polymer produced by other methods.

The time required to arrive at a single continuous phase, i.e. to complete the gelling depends to a large extent on the temperature of the liquid curing medium and the thickness of the coating to be gelled. Thus, when a 1 mm thick coating of a plastisol of a vinyl polymer consisting substantially wholly of vinyl chloride units on a hollow metal former is immersed in a bath of glycerol maintained at a temperature of 180° C, complete gelling is effected in a period of about two minutes. The gelling time also

varies to some extent with the amount of plasticiser incorporated in the plastisol and also on the heat losses which occur due to the nature of the material used for the former. Gelling times of less than 1 minute are possible in the method of the invention.

If desired, after gelling has been completed and the former removed from the liquid curing bath, traces of the liquid curing medium may be removed from the product. In the case of water miscible liquids, this can be achieved by washing the product with water.

The former can be constructed of any material such as porcelain or metal which has no surface porosity and the coating may be applied to the external or internal surface of the former.

A convenient way of forming a coating of plastisol of the external surface of a former is to dip the former into a bath containing the plastisol. The thickness of the coating obtained in this way depends on the time taken to withdraw the former after immersion in the plastisol, the temperature of the former and on the viscosity of the plastisol which itself depends on the temperature of the plastisol. When only single coating is required on the former the former is preferably warmed to a temperature of about 38° C. before being dipped in the plastisol. Preferably, the thickness of the coating is of the order of one millimeter.

Alternatively, the dispersion of vinyl polymers may be introduced into a hollow former, the internal surface of which is formed in the shape of the article to be produced, and the excess dispersion poured out of the former by inversion thereof. When the internal surface of a former is coated the gelling may be effected in the liquid bath either with or without direct contact between the coating and the liquid curing medium.

If the plastisol is heated to a temperature of about 100° C a semisolid is formed. This effect is normally referred to as pre-gelling. This pre-gelling effect may be utilised to form several layers of the plastisol on the former before complete gelling is effected. The pre-gelling effect can be achieved either by immersing a former coated with the plastisol into a liquid bath heated to a temperature of about 100° C or, by pre-heating the former itself to a suitable temperature. When multi-layers are desired, the former is first dipped in the normal way then immersed in the liquid bath maintained at a temperature of about 110° C to pre-gel the coating and is then again dipped into the plastisol to form a further coating on the former. Several coatings can be applied in this way until the desired thickness is achieved. When the desired thickness has been achieved the coated former can then be immersed in the liquid curing

medium at a temperature above 150° C in order to effect complete gelling of the coating to form a single uniform phase of plasticised resin. When the former itself is preheated the amount of pre-gelling which occurs and therefore the thickness of the coating formed depends upon the temperature of the former, the higher the temperature, the greater the amount of pre-gelling which occurs.

The invention also includes the production of protective clothing, especially gloves, provided with an inner lining. In the preferred form of the present invention the lining is fabricated from loose fibres which are readily formed into any desired shape and are subsequently set in that shape by the outer layer of polyvinyl chloride which additionally forms a base for the fibres.

According to one embodiment of the present invention, therefore, an article of protective clothing, especially a glove, is provided which comprises an inner layer or lining of individual natural or synthetic fibres fabricated into the general shape of the article and held in that shape by an outer layer of plasticised polyvinyl chloride which, prior to gelling, has partially impregnated the inner layer or lining whereby the fibres are held in a fabricated layer.

In the manufacture of the preferred lined article according to the present invention a former is dipped into a bath of plastisol, is slowly withdrawn and, whilst the plastisol is still in a liquid state, has applied thereto a coating of individual fibres substantially to cover the exposed plastisol whereafter the article is immersed in a liquid bath, heated to a temperature of at least 150° C for a time sufficient to effect complete gelling of the plastisol and the article then withdrawn and washed to remove any of the liquid adhering thereto and, finally, the article removed from the former, dried and turned inside out so that the fabricated layer of fibres constitutes an inner layer or liner for the article.

The individual fibres may be synthetic, such as cellulose acetate or may be natural fibres such as sheeps wool and asbestos. The fibrous material may be applied to the coated former in any convenient way as, for example, by passing the former through a chamber wherein the fibres are dispersed and kept circulating by means of a fan or blower, or by spraying with a spray gun. Advantageously, the application of fibres is carried out in an electrostatic field in order to impart a more or less uniform orientation to the individual fibres. The fibres may also be sprayed on to the coated former in the form of a flock. Preferably, the fibres are of short length, e.g. 0.3 millimeters.

In an alternative method according to the invention the former may be wetted or given a coating of adhesive whereafter a layer of

individual fibres is applied to the former to constitute an inner layer or lining, subsequent to which the article is dipped into a bath of plastisol and then immersed in the liquid curing bath as stated above.

A preformed lining, such as knitted fabric produced in the shape of the article, may also be used in the method of the present invention. The preformed lining is placed on the former and is then dipped into a bath of the plastisol and the plastisol coating gelled in the manner described above. An unlined article, such as a glove, produced according to the invention can, of course, be lined by applying an adhesive to the inner surface thereof and then applying individual fibres of natural or synthetic material to the surface so that they adhere thereto and form an inner lining. This is conveniently achieved by turning the article inside out before the adhesive is applied to the surface.

The compounds used to make up the liquid curing bath or the pre-gelling bath are preferably those which are substantially inert with respect to the ingredients of the plastisol. Compounds which have a marked solvent or chemical action on any of the ingredients should be avoided. When a lining is applied, compounds which chemically react with the lining, especially when this is of a synthetic material should not be used as ingredients of the liquid medium. Thus, when cellulose acetate fibres are used, the liquid bath should not contain aniline. The compounds forming the liquid bath should have boiling points above 150° C and preferably above 200° C. The compounds need not be liquid at all temperatures but may, for example, be solid at ordinary temperatures so long as they are liquid over the range of temperature needed for gelling of the plastisol. When any of the compounds used to form the liquid baths have flash points e.g. ethylene glycol care should be taken to avoid local overheating in order to avoid the combustion of the liquid bath.

It has further been found that articles manufactured from plastisols of polyvinyl chloride by the method of the present invention using ethylene glycol, glycerol or triethanolamine as the liquid bath for effecting the complete gelling of the plastisol possess an elastic recovery greatly in excess of that normally possessed by polyvinyl chloride sheeting and, in some cases, approaching that of rubber. With these liquids it is also found that they possess excellent flexibility. When manufacturing gloves according to the invention, therefore, it is preferred to use as the liquid bath ethylene glycol, glycerol or triethanolamine.

After the plastisol coating has been completely gelled, talc may be applied to the exposed surface of the article before removing it from the former. Such application of

talc facilitates removal of the article from the former. When the article is turned inside out by the act of removal from the external surface of a former, as is the case with gloves which do not include a prefabricated lining, the talc coated surface becomes the inner surface of the article after removal from the former.

The invention will be further illustrated by reference to the following Examples:

EXAMPLE I

PRODUCTION OF A LINED GLOVE

A metal former was heated to 38° C and then immersed in a plastisol of polyvinyl chloride, the immersion being effected over a period of twenty seconds. The plastisol used was a dispersion of 100 parts by weight of polyvinyl chloride in 100 parts by weight of di-*isooctyl* phthalate as plasticiser and including a total of 6 parts by weight of calcium zinc soap and an epoxidised oil as stabiliser. Such a plastisol is manufactured by Vinatex Ltd. Devonshire Road, Carshalton, Surrey, England and sold under the Trade name "Vinatex" (Registered Trade Mark) paste, reference PNT50/1798. The former was left immersed in the paste for a period of ten seconds and was then withdrawn slowly over a period of six minutes. The coated former was then passed through a field of charged fibres of cellulose acetate having an average fibre length of 0.3 millimeters until the coating on the former was covered with a layer of flock. The field was produced by means of a 40KV Flockstat (Registered Trade Mark) Power Unit.

A bath of glycerol was heated to and maintained at a temperature of 170° C. The coated former was immersed in the heated glycerol bath and was left immersed therein for a period of two minutes. The former was then removed from the glycerol bath, washed in running water and the resulting plasticised polyvinyl chloride glove removed from the former, the glove being turned inside out by the action of removal. The glove so formed was found to have excellent flexibility and good recovery from extension.

EXAMPLE II

PRODUCTION OF AN UNLINED GLOVE FROM POLYVINYL CHLORIDE

A metal former was heated to 38° C and was then immersed in the polyvinyl chloride plastisol of Example I, the immersion being effected over a period of twenty seconds. The former was left immersed in the paste for a period of ten seconds and was then slowly withdrawn, the withdrawal being effected over a period of six minutes. Further procedure was then identical with that set forth in Example I except that no lining was applied. A similar but unlined product to that produced in Example I was obtained.

EXAMPLE III

PRODUCTION OF A GLOVE USING A FABRICATED LINING

A knitted fabric lining was placed over a metal former and the former then immersed in the plastisol of Example I, the immersion being effected over a period of twenty seconds. The former was left immersed in the plastisol for a period of ten seconds and was then slowly withdrawn over a period of eight minutes.

A coating of the plastisol was formed thereby on the knitted fabric lining. The former and coated fabric lining were then immersed in a bath of glycerol maintained at 180° C and kept immersed in the bath for a period of three minutes. The former and lining were then removed and the glove so formed washed in running water and then removed from the former. The glove showed good flexibility and elastic recovery.

EXAMPLE IV

A porcelain former in the shape of a glove was heated to a temperature of 38° C and immersed in the plastisol of Example I containing a white pigment, the immersion being effected in twenty seconds. After ten seconds, the former was withdrawn at the rate of 2" a minute. The coated former was then immersed for two minutes in a bath of ethylene glycol maintained at a temperature of 170° C. A product of good flexibility and recovery from extension was obtained.

EXAMPLE V

A metal former in the shape of a glove was heated to approximately 150° C and was then immersed in the plastisol of Example I containing a red pigment. The immersion was effected in twenty seconds and after thirty seconds the former was withdrawn at the rate of 4" a minute. The coated former was then left clear of the plastisol for a period of thirty seconds and then again immersed in the plastisol over a period of twenty seconds, and, after a further period of ten seconds, withdrawn at the rate of 2" a minute. The coated former was then immersed for two and a half minutes in a bath of glycerol maintained at a temperature of 170° C and then withdrawn. The resulting product was a thick, unlined glove suitable for industrial use.

EXAMPLE VI

A metal former in the shape of an over-shoe was heated to 130° C and was immersed in the plastisol of Example I. The immersion was effected in twenty seconds. After a period of twenty seconds, the former was withdrawn at the rate of 2" a minute and then immersed for two minutes in a bath of glycerol maintained at a temperature of

170° C, and then withdrawn. The article so formed was a flexible overshoe.

EXAMPLE VII

5 A porcelain former in the shape of a glove was heated to 38° C. and immersed in a bath of the plastisol of Example I. The immersion was effected over a period of twenty seconds. After ten seconds the former was withdrawn at the rate of 2" per minute.
10 The coated former was then immersed in a bath of glycerol maintained at a temperature of 160° C. Complete gelling was found to have occurred after six minutes. When the procedure was repeated using a hollow
15 metal former, the time required for complete gelling was reduced from six minutes to two and a half minutes.

EXAMPLE VIII

20 A porcelain former in the shape of a glove was heated to 110° C and then immersed in the plastisol of Example I containing red pigment. The immersion was effected in twenty seconds. After thirty seconds the former was withdrawn at the rate of 2" per
25 minute. The coated former was then immersed for two minutes in a bath of glycerol maintained at a temperature of 170° C.

EXAMPLE IX

30 A thin metal former shaped internally in the form of a teat was filled with the plastisol of Example I and then inverted to allow surplus plastisol to drain off. The coated former was then immersed completely in a bath of glycerol maintained at 170° C for
35 thirty seconds by which time gelling was complete. The former was cooled by immersion in cold water and the teat removed.

EXAMPLE X

40 Fifty parts by volume of a powder consisting of a vinyl chloride/vinyl acetate copolymer containing 18% of vinyl acetate units (e.g. as sold under the reference GEON 425 (Registered Trade Mark) by British Geon Limited, Devonshire House, Piccadilly,
45 London, W.1) are thoroughly mixed with fifty parts by volume of dibutyl phthalate. An aluminium tube bearing external impressions of a name was coated with the resulting plastisol, and the coated tube
50 completely immersed in glycerol maintained at 175° C for a period of two minutes by which time gelling was complete. The tube was cooled by immersion in cold water, talc applied to the gelled coating and the
55 gelled coating removed. The gelled coating showed excellent flexibility and reproduction of the impression on the tube.

EXAMPLE XI

60 50 parts by volume of an organosol containing 100 parts polyvinyl chloride, 40 parts

plasticiser in the form of an adipate, 3 parts of a barium/cadmium stabiliser, 0.5 parts epoxidised oil, 0.5 parts viscosity depressant and a black pigment (such as that sold under the name "Spraying Paste PSP/80" by
65 Vinatex Limited) were mixed with 25 parts of thinners containing 25% white spirit, and the remainder xylene with a small amount of ketone and hydrocarbon (e.g. that sold under the reference ST6 by Vinatex Limited). A
70 metal float (level control) was sprayed with the mixture to form a coating thereon and the coating allowed to dry in air for three hours. The coated float was then immersed for two minutes in glycerol maintained at
75 170° C and cooled in water. The thickness of the coating formed can be varied by varying the amount of thinners employed.

EXAMPLE XII

80 One face of a metal sheet was coated (as by brushing or smearing) with a plastisol formed by intimately mixing 50 parts by volume of a powdered vinyl chloride/vinylidene chloride copolymer containing 2%
85 vinylidene chloride units, with 50 parts dibutyl phthalate. The coated sheet was then immersed in glycerol maintained at 170° C. Gelling was complete in two minutes after which time the coated sheet was cooled by
90 immersion in water. An adhesive may be first applied to the former if desired.

EXAMPLE XIII

A metal former in the shape of a tubular grip (e.g. for a tennis racquet) was dipped
95 into the plastisol of Example I and withdrawn at the rate of 2 inches per minute. The coated former was immersed for one minute in glycerol maintained at 175° C and then cooled by immersion in water. Talc was applied and the coating removed. The
100 grip thus produced showed good flexibility.

WHAT WE CLAIM IS:—

1. A process for the production of an article from or the coating of an article with vinyl polymers and copolymers containing at
105 least a major proportion of combined vinyl chloride which comprises applying to a former in the shape of said article one or more coating of a dispersion of at least one of said polymers or copolymers in a plasticiser
110 therefor and thereafter effecting complete gellation of said coating on said former by directly contacting said coating with a liquid medium maintained at a temperature at which
115 gellation of said dispersion takes place.
2. A process as claimed in claim 1 in which the vinyl polymers consist substantially wholly of polyvinyl chloride.
3. A process as claimed in claim 1 in which the vinyl polymers comprise a copolymer of
120 vinyl chloride and vinyl acetate.
4. A process as claimed in claim 1 in

- which the vinyl polymers comprise a copolymer of vinyl chloride and vinylidene chloride.
5. A process as claimed in claim 3 or 4 in which the copolymer contains at least 95% vinyl chloride.
6. A process as claimed in claim 1 in which the former is a porcelain former.
7. A process as claimed in any of the preceding claims in which the gelling temperature is in the range of from 150° C. to 200° C.
8. A process as claimed in claim 7 in which the gelling temperature is in the range of from 160° C. to 180° C.
9. A process as claimed in any of claims 1 to 8 in which the liquid medium comprises ethylene glycol, glycerol or triethanolamine.
10. A process as claimed in any of claims 1 to 9, in which the coating is applied to the former by immersing the former in a bath containing the dispersion of polymers in a plasticiser therefor.
11. A process as claimed in any of the preceding claims in which the former is heated to a temperature of substantially 38° C prior to being coated with the dispersion of polymers.
12. A process as claimed in any of claims 1 to 11 in which the former is heated to a temperature greater than 100° C prior to being coated with the dispersion of polymers whereby the coating formed is in a pregelled state.
13. A process as claimed in claim 12 in which the coated former is again coated with the dispersion of polymers to form a second coating thereof and the coated former then immersed in the liquid medium to effect complete gelling of the composite coating.
14. A process as claimed in claim 10 in which a plurality of coatings are formed on the former by consecutively immersing the former in the dispersion of polymers and then immersing the coated former in a liquid medium maintained at a temperature of substantially 100° C.
15. A process as claimed in any of the preceding claims in which, after applying the coating to the former and whilst said coating is still in a liquid state individual fibres of natural or synthetic material are applied to the said coating substantially to cover the exposed area of the coating on the former.
16. A method as claimed in claim 15 in which the individual fibres are applied to the coated former whilst the former is under the influence of an electro-static field.
17. A method as claimed in any of the preceding claims in which a preformed lining of knitted fabric in the form of article of protective clothing is placed on the former and the plastisol applied to said preformed lining.
18. A method as claimed in any of the preceding claims in which the article is an article of protective clothing.
19. A method as claimed in claim 18 in which the article is a glove.
20. An article of protective clothing comprising an inner layer or lining of individual natural or synthetic fibres fabricated into the general shape of the article and held in that shape by an outer layer of plasticised polyvinyl chloride which, prior to gelling, has partially impregnated the inner layer or lining whereby the fibres thereof are held in a pre-fabricated layer whenever produced by the process claimed in claim 1.
21. A method of manufacturing an article composed of vinyl-polymers substantially as hereinbefore described in any one of the examples.
22. An article composed of vinyl-polymers whenever produced by the process claimed in any of claims 1 to 20 or 21.

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